Low-loss dielectric mirror with ion-beam-sputtered TiO_2–SiO_2 mixed films

Shiuh Chao, Wen-Hsiang Wang, and Cheng-Chung Lee

Ion-beam-sputtered TiO_2–SiO_2 mixed films with 17% SiO_2 concentration were used as high-refractive-index layers in a multilayered-stack dielectric mirror. Experimental results indicated that total loss of the as-deposited mirror was 34% lower than that of the as-deposited conventional mirrors with pure TiO_2 films used as high-refractive-index layers. In addition, annealing reduced total loss of the mirrors. Although decreasing with an increasing annealing temperature, total loss of the conventional mirrors dramatically increased above ~200 °C annealing temperature, owing to increased scattering from an amorphous-to-crystalline phase transition in the TiO_2 films. In addition, total loss of the mirrors with the mixed films continuously decreased with an increasing annealing temperature up to 400 °C without the phase transition. Total loss was reduced 88% by means of decreasing absorption in the mixed films. Moreover, the annealed mirror with mixed films was better than both the as-deposited mirror and the conventional mirror with pure films in terms of laser-damage resistance. © 2001 Optical Society of America

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1. Introduction

Scattering loss and absorption loss of a dielectric mirror can cause lock in and lock-in growth phenomena in a ring-laser gyroscope. Previous investigations proposed that burn-in grating on a mirror, as produced by interference of the counter directional propagate beams by means of photochromic, photorefractive, or temperature-dependent-of-refractive-index effects, is the mechanism of lock-in growth phenomenon.[1,2] Scattering loss and absorption loss can also cause mirror damage in high-energy laser applications. Notably, in sensitive interferometer applications such as gravitational-wave detection, ultralow optical loss of the mirrors is required. For this application, Rempe *et al.*[3] reported on a dielectric mirror with 6 parts in 10^6 (ppm) total loss at 1064 nm. A dielectric mirror is composed of alternating high- and low-refractive-index thin films, normally with a quarter-wave optical thickness. For applications related to the visible-wavelength region, TiO_2 and Ta_2O_5 are the conventionally used materials for high-refractive-index thin films. In addition, SiO_2 and Al_2O_3 are frequently used as low-refractive-index thin-film materials. Ion beam sputtering, i.e., the conventional means of depositing these films,[4] produces films with improved qualities such as high density with a high refractive index and amorphous structure with a low scattering loss.[5] Although postdeposition annealing is frequently used to reduce absorption loss of the multilayer dielectric mirror, ion-beam-sputtered TiO_2 thin films are transformed from amorphous to polycrystalline anatase phase at ~200 °C annealing temperature.[6] Notably, the polycrystalline grain boundary and roughened surface of annealed TiO_2 film increases the scattering loss. Therefore increasing scattering from the appearance of the polycrystalline phase limits the ability of higher-temperature annealing for further reduction of the absorption loss of the films.

As is well known, TiO_2–SiO_2 mixed films have many advantages over conventional thin films. Previous investigations[7,8] indicated that adding SiO_2 into TiO_2 film to form TiO_2–SiO_2 mixed film prevents crystallization of the TiO_2 film. Adding different amounts of SiO_2 into TiO_2 film leads to a wide range of refractive-index tuning; the mixed film structure remains amorphous as well. These mixed films can therefore be used in applications such as refractive-index tuning, graded-index films, and rugate filters. A more recent study[9] revealed that TiO_2–SiO_2 mixed

S. Chao (schao@ee.nthu.edu.tw) and W.-H. Wang are with the Electrical Engineering Department, National Tsing Hua University, Hsin-Chu, Taiwan. C.-C. Lee is with the Institute of Optical Science, National Central University, Chung-Li, Taiwan.

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film deposited by the ion-beam-sputter method is low absorptive and amorphous and that the refractive index is allowed to be tuned and can sustain high-temperature annealing without crystallization. The crystallization temperature increases from ~200 °C for pure TiO_2 to ~250 °C for 5% SiO_2 mixed films, to ~300 °C for 9% SiO_2 mixed films, and beyond 400 °C for 17% SiO_2 mixed films. Absorption of the mixed films decreases with an increasing amount of SiO_2 and increasing annealing temperature. Therefore incorporating the ion-beam-sputtered TiO_2–SiO_2 mixed films into a dielectric multilayer stack to achieve low absorption and low scattering losses, i.e., low total loss, is feasible as implied by that same study.[9]

In this paper we use ion-beam-sputtered TiO_2–SiO_2 mixed films and SiO_2 films as the high-refractive-index layer and the low-refractive-index layer, respectively, for a multilayered-stack dielectric mirror. High-temperature annealing is performed on the mirrors. In addition, total loss and laser-damage resistance of the mirrors are compared with those of a conventional mirror in which pure TiO_2 is used as the high-refractive-index layers. This study also investigates whether the single-film properties of TiO_2–SiO_2 mixed films such as lower absorption, resistance to amorphous–crystalline phase transition and low scattering as reported in an earlier study,[9] would be preserved in the multilayered stack. Microstructural variations of the mirrors as they undergo high-temperature annealing are also discussed, as are interfacial problems.

2. Experiment

A. Multilayer-Stack Coating

The substrate material used in this study was Zerodur. The Zerodur substrates were polished to a flatness of 1/10λ and a roughness of 0.17 nm. All dielectric mirrors reported herein have 23 layers of alternating high- and low-refractive-index films. Each film has λ/4 optical thickness tuned at 632.8 nm. The low-refractive-index films were SiO_2, and the high-refractive-index films were TiO_2–SiO_2 mixed films with 17% SiO_2 concentration. The first layer adjacent to the substrate was the high-refractive-index film. A half-wave-thick SiO_2 layer covered the top of the multilayer stack. Mirrors with pure TiO_2 used as the high-refractive-index films were also constructed for comparison with the mirrors with TiO_2–SiO_2 mixed films. The correct time duration for sputtering λ/4-thick film was determined beforehand for each film. The sputtering time was varied to control the films' thicknesses in the multilayered stack.

All films were deposited by ion beam sputtering with a 2.5-cm Kaufman-type ion source. The sputtering parameters were 1-kV beam voltage and 50-mA beam current. A plasma bridge neutralizer was used to neutralize the beam. Oxygen gas of 5×10^{-4} mbar partial pressure was then fed into the sputtering chamber with 4×10^{-4} mbar of argon for reactive sputtering of all films. A 4*N*-purity SiO_2 sputter target was used to deposit SiO_2 films. A 4*N*-purity TiO_2 target was also used to deposit pure TiO_2 films. Next, a small piece of silicon wafer with fixed area was attached to the titanium target to deposit TiO_2–SiO_2 mixed films. The SiO_2 concentration in the mixed films was 17% as determined by Rutherford backscatter spectroscopy. SiO_2 concentration of 17% in the mixed films was selected on the basis of a previous study.[9] Results in the previous study[9] indicated that mixed films with 17% SiO_2 concentration could sustain a higher annealing temperature than those with a lower SiO_2 concentration yet still have a sufficiently high refractive index. Details of the deposition conditions and characteristics of the pure TiO_2 films and TiO_2–SiO_2 mixed films can be found in Refs. 6, 9, and 10.

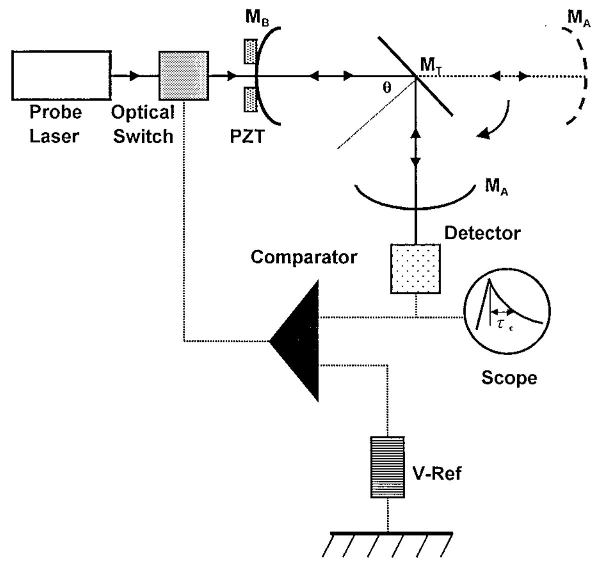

Fig. 1. Schematic diagram of cavity ring-down setup.

High-temperature annealing on the mirrors was performed in a furnace with atmospheric environment. The annealing time was 24 h for all mirrors. Mirrors were carefully packed in a glass container to avoid contamination during annealing. In earlier studies[6,9] ion-beam-sputtered pure TiO_2 film underwent a phase transition from amorphous to crystalline near 225 °C annealing for 24 h, and 17% TiO_2–SiO_2 mixed films underwent same phase transition beyond 400 °C annealing for 24 h. To investigate whether the same annealing effects would occur in the multilayered-stack mirrors, we allow the multilayered-stack mirrors with pure TiO_2 films to go through annealing at 180 and 225 °C and the mirrors with TiO_2–SiO_2 mixed films to go through 200, 300, and 400 °C annealing, respectively.

B. Cavity Ring-Down Measurements

A cavity ring-down loss meter was used to measure the mirror reflectance within an accuracy of several parts in 10^6. Figure 1 schematically depicts the measurement setup. Standard mirrors M_A and M_B

Table 1. Comparison of Mirror Performance

Sample Number	SiO_2 (%) Fraction	Annealing Temperature (°C)	Total Loss (ppm)	Total Loss Change (%)	rms Surface Roughness (nm)	Damage (kJ/cm^2) Threshold[a]
1	0	As deposited	430 ± 12	0	0.18	2.80
2	0	180	142 ± 10	−67 ± 2	0.19	6.12
3[b]	0	225	216[c]	−50	0.55[c]	Immediate damage
4	17	As deposited	282 ± 36	0	0.16	4.27
5	17	200	72 ± 7	−74 ± 2	0.17	>6.37
6	17	300	41 ± 12	−85 ± 4	0.16	>6.37
7	17	400	34 ± 11	−88 ± 4	0.17	>6.37

[a]The highest available test energy density was 6.37 kJ/cm^2.
[b]Microsize cracks developed for 225 °C.
[c]Measured at area where there were no microsize cracks.

were initially inserted to form a linear cavity. With the optical switch turned on, the piezoelectric transducer adjusted the cavity length to achieve resonance. When the transmitted intensity reaches a certain level as resonance occurs, the optical switch is turned off through a comparator. The transmitted intensity then decays exponentially, as observed with an oscilloscope. Assuming that no distributed loss occurs in the cavity, the characteristic decay time (τ_c) is related to the mirrors' reflectance R_A and R_B as shown in Ref. 11:

$$\tau_c = l/C[1 - (R_A R_B)^{1/2}],$$

where l is the cavity length, C is the speed of light, and R_A and R_B are the mirror reflectances. The reflectance product $R_A R_B$ can thus be obtained.

Next the test mirror M_T was placed in the cavity, as shown in Fig. 1. The polarization state of the incident light and the folding angle Θ of the instrument were adjusted so that they were consistent with the designed polarization and angle of incidence of the test mirror. The decay time of the cavity with mirror $M_A M_B M_T$ could be measured, allowing us to obtain the reflectance of the test mirror.

Here the total loss of a mirror is defined as scattering loss plus absorption loss. We measured transmittance (T) of the mirrors by taking the ratio of the transmitted power to the incident power to a high accuracy. Reflectance (R) of the mirrors was then measured by use of the cavity ring-down loss meter with a 632.8-nm He–Ne laser source as described above. Finally, total loss of the mirror at 632.8 nm wavelength was obtained as $1 - R - T$.

C. Laser-Damage-Resistance Test

The laser-damage test was conducted by means of focusing a fiber-guided 1064-nm pulse output from a Nd:YAG laser onto a mirror surface. The pulse width was 6 ms. The focus spot size on the mirror surface was 1 mm in diameter. The maximum pulse energy available was 50 J. Here the energy density is defined as the pulse energy divided by the focal area. The maximum available energy density on the mirror surface was therefore 6.37 kJ/cm^2.

The test area could be viewed, and the reflectance of the test area was monitored *in situ* by a low-power cw He–Ne laser. Pulses with different energies were given to different areas. Only one pulse was given to each test area. Here we considered the mirror to be damaged once the reflectance of the test area (as monitored by a low-power He–Ne laser) was varied by the Nd:YAG laser pulse.

D. Surface Roughness Measurement

Surface roughness of the mirrors was measured by atomic force microscopy. The atomic force microscope used here was Nanoscope III purchased from Digital Instruments. In addition, a large-scale tapping mode was used, in which the scanning area was 1 μm × 1 μm, the scanning speed was 1 μm/s, and each scan contained 256 × 256 sample points. The precision of the surface roughness, as determined in our laboratory, was 0.04 nm.

3. Results and Discussion

A. Total Loss and Laser Damage of the Multilayer-Stack Mirror

Several mirrors with pure TiO_2 used as the high-refractive-index films and pure SiO_2 used as the low-refractive-index films were made with the exact same deposition conditions. Total loss of these as-deposited mirrors was then measured on several different spots for each mirror. The average value and rms deviation of these measurements were 430 ± 12 ppm as listed in the first row of Table 1 and labeled as sample 1. The rms deviation included sample-to-sample and spot-to-spot variations. These mirrors were then subjected to annealing at different temperatures for 24 h. After annealing, total loss of the mirror that was subjected to 180 °C annealing was reduced to 142 ± 10 ppm; the rms deviation used here included only spot-to-spot variation on the same mirror. Microsize cracks on the mirror surface were observed after annealing for the mirror that was subjected to 225 °C annealing. Only a small area on this mirror could be found where no cracking occurred. Total loss of this area was measured to be 216 ppm. Surface roughness of this area was 0.55 nm. In addition, serious cracking and peeling occurred after annealing for mirrors that were subjected to annealing above 225 °C. Notably, the total

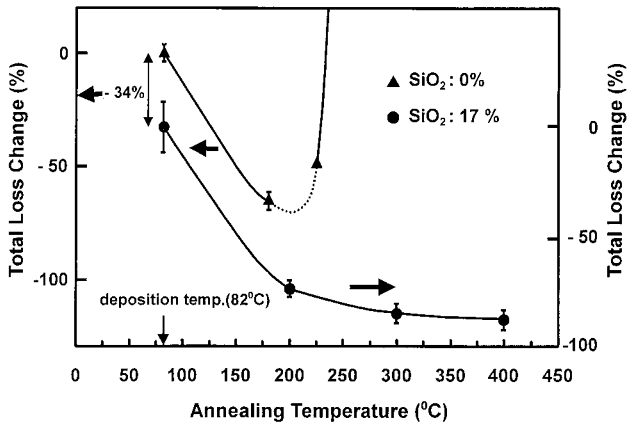

Fig. 2. Percentage change of total loss versus annealing temperature. Triangles, mirror with pure TiO_2 film; circles, mirror with TiO_2–SiO_2 mixed film.

loss of these mirrors was excessively large, and thus that cavity photon lifetime was too short to measure. Damage threshold was found to be 2.80 kJ/cm^2 for the as-deposited mirror, 6.12 kJ/cm^2 for the mirror that was subjected to 180 °C annealing, and immediate damage for mirrors that were subjected to 225 °C and higher annealing temperatures.

Several mirrors with mixed TiO_2–SiO_2 as the high-refractive-index films and pure SiO_2 as the low-refractive-index films were made under the exact same conditions. The SiO_2 content in the mixed films was 17%. Average total loss and rms deviation of these as-deposited mirrors was 282 $\pm$ 36 ppm as listed in the fourth row of Table 1, labeled sample 4. These mirrors were then subjected to annealing temperatures of 200, 300, and 400 °C, respectively. Total loss was measured on various spots of each mirror after annealing, in which Table 1 lists the average values and rms deviations for each mirror. According to this table, total loss decreased when the annealing temperature was increased. Notably, surface roughness of the mirrors remained unchanged after annealing. The damage threshold of the as-deposited mirror was 4.27 kJ/cm^2. No damage could be observed for mirrors that were subjected to 200, 300, and 400 °C annealing, respectively. The maximum energy density per pulse for damage test was 6.37kJ/cm^2.

Figure 2 summarizes the percentage change in total loss versus annealing temperature for mirrors with pure TiO_2 films (triangles) and mirrors with TiO_2–SiO_2 mixed films (circles). The first point on each curve denotes the as-deposited mirror without annealing. The substrate temperature during deposition was 82 °C. Total loss of the as-deposited mirror was taken as a standard for comparing with the annealed mirrors for each curve, respectively.

Figure 2 also indicates that total loss of mirrors with pure TiO_2 films decreased with an increasing annealing temperature and, then, markedly increased at ~200 °C. Total loss of the mirrors with mixed TiO_2–SiO_2 films decreased with an increasing temperature up to 400 °C. Moreover, total loss of the as-deposited mirror with the mixed films was 34% lower than that of the as-deposited mirror with pure films.

Laser-damage results in Table 1 are correlated with the trend of total loss variation in Fig. 2. A higher damage threshold can be obtained for mirrors with mixed films annealed at a higher temperature. According to our results, laser damage of the mirrors was catastrophic and abrupt. Restated, morphology and reflectance suddenly and drastically changed as the laser energy reached the threshold point. However, no change occurred immediately below the threshold.

Notably, laser-damage-threshold testing was conducted with a Nd:YAG laser at a wavelength of 1064 nm on the high-reflectance mirrors that were designed for 632.8 nm. The damage-threshold values presented here are meaningful only for comparison between mirrors with different SiO_2 concentrations and different annealing temperatures. Therefore these values should not be compared with those of the mirrors designed for 1064 nm and tested with a 1064-nm laser.

B. Microscopic Variations of the Multilayer-Stack Mirror with Annealing

The relationship between the multilayered mirror and single-film characteristics presented in Refs. 6 and 9 were investigated. Tables 2 and 3 list the properties of single pure TiO_2 films and the properties of single 17% TiO_2–SiO_2 mixed films from Refs. 6, 9, and 10. Refractive index n and extinction coefficient k that were deduced from the transmission spectra at 632.8, 550, and 450 nm of the single films are given. Here I/I_0 is the relative x-ray diffraction intensity of the (101) peak for the TiO_2 anatase phase, serving as an indicator of the extent of amorphous-to-polycrystalline transition caused by different annealing temperatures. In addition, λ_c is the cut-off wavelength, at which, the transmittance of the film decreased to zero, serving as a general indicator of the extent of absorption in the UV–visible range. Notably, a lower λ_c implies a lower absorption.[6] Detailed discussions of Tables 2 and 3 and their schematic illustrations can be found in Refs. 6, 9, 10.

Comparing Fig. 2 with Table 2 and Fig. 3 of Ref. 6 reveals that the decreasing total loss of the mirror in which a pure TiO_2 film was used as the high-index layer correlated with the decreasing extinction coefficient of the single pure TiO_2 film. In addition, the significant increase of the total loss at ~200 °C for the mirror correlated with the phase transition of the single pure TiO_2 film.

Comparing Fig. 2 with Table 3 and Fig. 2 of Ref. 9 reveals that the decrease of the total loss of the mirror, in which the 17% TiO_2–SiO_2 mixed film was used as the high-refractive-index layer, correlated with the decreasing extinction coefficient of the single 17% TiO_2–SiO_2 mixed film.

On the basis of the above observations, we can infer

Table 2. Refractive Index (n), Extinction Coefficient (k), Relative X-Ray Diffraction Intensity I/I_0 for Anatase (101) Peak, Cut-Off Frequency (λ_c), and rms Surface Roughness of the Pure TiO_2 Film[a]

Annealing Temperature (°C)	632.8 nm		550.0 nm		450.0 nm				
	n	k	n	k	n	k	I/I_0	λ_c (nm)	rms Surface Roughness (nm)
Before annealing	2.50	0.0001	2.54	0.0003	2.66	0.0013	0	327.6	0.12
150	2.50	$<10^{-4}$	2.54	0.0002	2.66	0.0012	0	325.0	0.15
200	2.50	$<10^{-4}$	2.54	0.0001	2.66	0.0010	0	322.4	0.14
225	2.49	0.0001	2.53	0.0002	2.65	0.0011	20	319.2	0.84
250	2.48	0.0004	2.52	0.0007	2.64	0.0023	65	316.5	1.34
275	2.48	0.0007	2.52	0.0012	2.64	0.0040	100	313.8	1.72
300	2.48	0.0015	2.52	0.0025	2.64	0.0052	100	313.5	3.26
350	2.48	0.0018	2.52	0.0027	2.64	0.0062	100	313.5	3.30

[a]Refs. 6 and 10.

Table 3. Refractive Index (n), Extinction Coefficient (k), Relative X-Ray Diffraction Intensity I/I_0 for Anatase (101) Peak, Cut-Off Frequency (λ_c), and rms Surface Roughness of 17% SiO_2 Content TiO_2–SiO_2 Mixed Film[a]

Annealing Temperature (°C)	632.8 nm		550.0 nm		450.0 nm				
	n	k	n	k	n	k	I/I_0 (%)	λ_c (nm)	rms Surface Roughness (nm)
Before annealing	2.33	$<10^{-4}$	2.37	$<10^{-4}$	2.48	0.0006	0	314.1	0.16
200	2.32	$<10^{-4}$	2.36	$<10^{-4}$	2.47	0.0005	0	309.6	0.17
300	2.30	$<10^{-4}$	2.34	$<10^{-4}$	2.44	0.0003	0	307.6	0.17
400	2.28	$<10^{-4}$	2.32	$<10^{-4}$	2.41	0.0002	0	304.1	0.16
450	2.25	0.0004	2.29	0.0005	2.36	0.0009	0	303.4	0.30
500	2.21	0.0010	2.24	0.0012	2.32	0.0032	0	—	4.75
550	—	—	—	—	—	—	10	—	—

[a]Refs. 9 and 10.

that the properties of the ion-beam-sputtered single TiO_2 films and single TiO_2–SiO_2 mixed films were preserved in the multilayered-stack mirrors in which they were used as the high-refractive-index film.

Therefore variation of the microscopic properties of the multilayer stack mirrors with annealing temperature can be deduced from that of the single-layer film as follows. For a mirror with pure TiO_2 used as the high-refractive-index layer, below ~200 °C, absorption of the amorphous TiO_2 films in the multilayer stack mirror decreases with an increasing annealing temperature, because of oxidation. Total loss of the multilayer stack decreases, owing largely to decreasing absorption of the TiO_2 films. At ~200 °C, crystallization of the TiO_2 film occurs, the amorphous structure changes to polycrystalline anatase phase, and the polycrystalline grain boundary and roughened surface cause the scattering to increase markedly. Interestingly, total loss of the multilayer stack significantly increases, owing largely to scattering from the polycrystalline TiO_2 anatase phase. For a mirror with 17% TiO_2–SiO_2 mixed films used as the high-refractive-index layer, absorption of the amorphous TiO_2–SiO_2 mixed films in the multilayer-stack mirror decreases with an increasing annealing temperature, owing to oxidation. Moreover, adding SiO_2 to TiO_2 retards the formation of polycrystalline phase, thus enabling the mixed films to undergo a higher annealing temperature without crystallization. Therefore total loss of the multilayer stack mirror monotonically decreases with an increasing annealing temperature, and a decrease of the total loss of the mirror with mixed films is largely due to a decreasing absorption of the mixed films. A decrease in total loss is nearly complete at 400 °C, as indicated in Fig. 2. This observation implies that 24-h annealing at 400 °C enhances the oxidation of the mixed films in the multilayer stack, nearly to saturation.

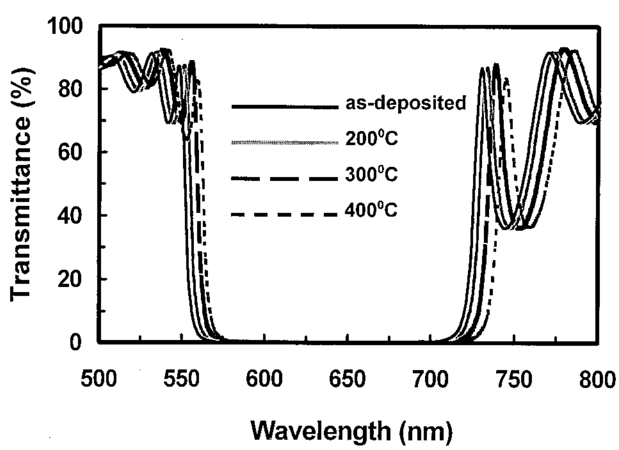

Fig. 3. Transmission spectra of the as-deposited mirror (solid black curve), mirrors after 200 °C (solid gray curve), 300 °C (long-dashed curve), and 400 °C (short-dashed curve) annealing.

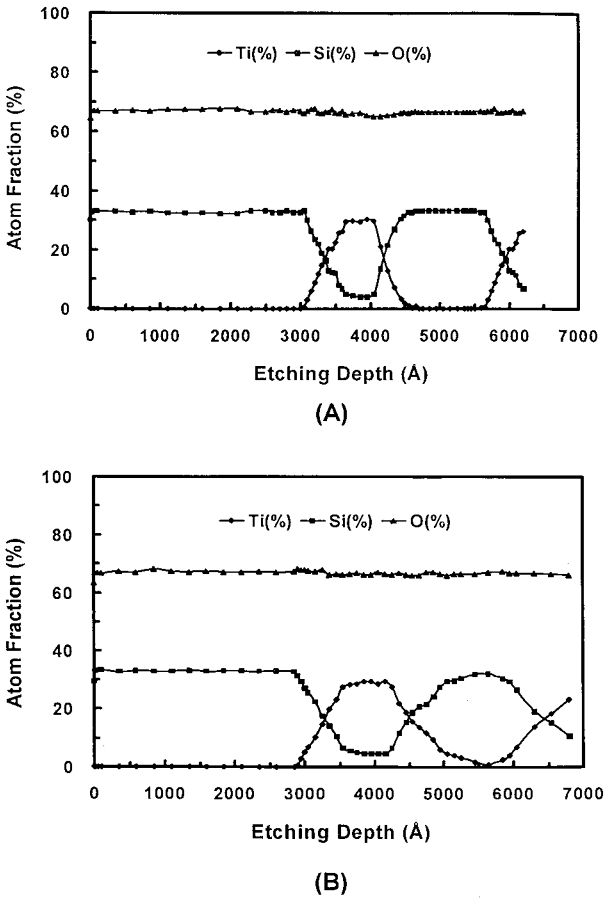

Fig. 4. Electron Spectroscopy for Chemical Analysis concentration depth profiles of (A) the as-deposited mirror and (B) mirror after 400 °C annealing. Diamonds, titanium; squares, silicon; triangles, oxygen.

C. Transmission Spectra and Concentration Depth Profiles of the Mirror

Figure 3 illustrates the transmission spectra of the mirrors with 17% TiO_2–SiO_2 mixed films before and after 200, 300, and 400 °C annealing, respectively. The mirrors were quarter-wave stacks designed for 632.8-nm wavelength. This figure reveals that the high-reflection band shifts toward a longer wavelength as the annealing temperature increases. Table 3 indicates that the refractive index at the 632.8-nm wavelength of the single 17% TiO_2–SiO_2 mixed film decreases with an increasing annealing temperature. If we assume that the multilayered stack consists of homogeneous films with a steplike refractive-index distribution along the depth, optical thickness of the mixed films should decrease with an increasing annealing temperature, and the high-reflection band of the mirror should therefore be shifted to a shorter wavelength, thus contradicting the observation in Fig. 3. For fuller understanding of the contradiction, Electron Spectroscopy for Chemical Analysis is used here to measure the concentration depth profiles of the top three layers of the multilayer stacks before and after 400 °C annealing. The topmost layer was a half-wave-thick SiO_2 film, the second layer was a quarter-wave-thick 17% TiO_2–SiO_2 mixed film, and the third layer was a quarter-wave-thick SiO_2 film. Figure 4(A) illustrates the titanium, silicon, and oxygen depth profiles before annealing, and Fig. 4(B) shows those profiles after 400 °C annealing. The ordinates of Fig. 4 are calibrated for the argon-ion etching depth and should not be taken as the absolute film thickness.

According to Fig. 4, the as-deposited mirror did not have well-separated layers as expected. The interface between different layers has a graded concentration distribution, since the ion-beam-sputter process is a higher-energy deposition process than deposition processes such as evaporation or ordinary sputter. The atoms that impinge the substrate in the ion-beam-sputter process normally have kinetic energies significantly higher than in the evaporation process. The surface temperature is therefore substantially higher in the ion-beam-sputter process than in the evaporation process, implying that the *in situ* diffusion phenomenon is more significant in the ion-beam-sputter process than in the evaporation process. Therefore we believe that the interface between layers is interdiffused and not well separated.

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